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HYDROGEN EMBRITTLEMENT OF A CYCLICALLY DEFORMED HIGH STRENGTH AL ALLOY

RENSSELAER POLYTECHNIC INSTITUTE
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HIGH STRENGTH Al ALLOY

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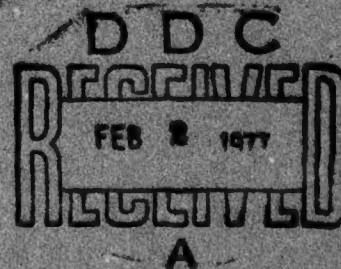Materials Engineering Department
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Abstract

High cycle fatigue experiments have been performed on a
7075 Al alloy, principally in the T6 temper in dry air, distilled
water $0.5\underline{N}$ NaCl and $0.5\underline{N}$ Na_2SO_4 solutions as functions of pH,
cathodic charging and catalyst poisoning of the hydrogen
evolution reaction. All aqueous solutions appreciably lowered
fatigue resistance with Cl^- ion producing the greatest reduction
in resistance and $SO_4^=$ ion behaving essentially in the same
manner as distilled water.   Under cathodic charging conditions
fatigue resistance is significantly reduced and both Cl^- and $SO_4^=$
solutions produce similar fatigue lives. Acid solutions reduce
fatigue resistance to a greater degree then do basic solutions
of equivalent corrosivity and a catalyst poison (As) added to Cl^-
solutions reduces fatigue resistance relative to neutral Cl^-
solutions. Fractography of specimens fatigued in aqueous
environments shows that a significant amount of cleavage and
quasi-cleavage occurs, the extent of these features being apparently
a function of available hydrogen to the alloy free surface and to
the tips of growing cracks. On the basis of these observations,

it is suggested that corrosion fatigue of 7075 alloy is essentially

a hydrogen embrittlement phenomenon where the low diffusivity of

hydrogen is counterbalanced by the fact hydrogen need only be

present in the alloy free surface for crack initiation and in

the plastic zone of growing cracks for propagation.

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Introduction

Environmentally accelerated fatigue crack initiation and
growth have been the subject of a considerable amount of recent
research[1-9]. Mechanisms for this phenomenon, particularly in
chloride containing solutions, have included active dissolution
of active slip bands and surface energy reduction at crack tips
due to preferentially adsorbing environmental species[4-6]. The
possibility of a hydrogen embrittlement phenomenon was also
suggested as one of several possibilities to explain reduced
fatigue resistance of aluminum alloys exposed to water vapor[7].

In previously reported work it was shown that corrosion
fatigue crack initiation of 7075 Al alloys may be associated with
hydrogen, generated by corrosion at non-metallic inclusions which
intersect the alloy surface[8]. Similarly, initial crack growth
was associated with hydrogen generated in an acid environment in a
growing crack which acts as a crevice. Additionally it was shown
that these effects are due to hydrogen contained in the alloy
lattice, in contrast to surface energy reduction at a crack tip,
since fatigue resistance of pre-charged specimens could be improved·
by a post-charging heat-treatment which effectively "baked" the

hydrogen out of the alloy [9] . It was also shown that simultaneous

cathodic charging of a cyclically stressed high purity analogue

of the alloy (Al 5Zn 2.5Mg) resulted in very low fatigue

resistance with crack surfaces showing a cleavage-type appearance.

Greater reductions in fatigue resistance and large numbers of

secondary cracks were observed for more negative cathodic

potentials [9] .

The results of the present study are part of a continuing

effort to assess and understand the roles of hydrogen on the

fatigue resistance of high-strength precipitation-hardened

aluminum alloys.

Experimental Procedure

Axial fatigue experiments have been performed on cylindrical

cross section (2.2mm) specimens of a commercial 7075 alloy in

the T6 heat-treatment in dry air, 0.5$\underline{N}$ NaCl (as f(pH)), 0.5$\underline{N}$

Na_2SO_4 and in 0.5$\underline{N}$ NaCl solution with addition of 10ppm As. For

some experiments specimens were cathodically charged to produce

hydrogen. Additionally some experiments were performed on

specimens in the T73 condition (which has been reported to show

superior SCC resistance) in dry air and in 0.5$\underline{N}$ NaCl solutions.

Fatigue loading was in the tension-tension mode with a constant

applied mean stress of 172 MPa ($\approx \sigma_y/3$) under load control of

30 Hz. The environmental test apparatus has been previously

described [8] . Prior to testing, specimen surfaces were mechanically

polished to 3μm diamond paste and subsequently electropolished.

Solutions were aerated and recirculated from an external reservoir during testing. Fracture surfaces and the specimen free surfaces for each case were examined by optical microscopy and SEM.

Results

Figure 1 shows the fatigue behavior of the alloy in air, distilled water, $0.5\underline{N}$ Na_2SO_4 and $0.5\underline{N}$ NaCl and shows that, except at relatively low applied stresses, H_2O and Na_2SO_4 solutions show approximately the same fatigue behavior. Exposure to NaCl solution during cyclic stressing, on the other hand, results in very inferior fatigue resistance with the 10^7 cycle fatigue life corresponding to an applied cyclic stress of only about 35 MPa. A static loading test in NaCl, at an applied stress of about 320 MPa (a $\sigma_{mean} + \sigma_{cyclic\ max}$ of this magnitude would result in $N_f = 3 \times 10^4$ cycles) showed no evidence of cracking for a period greater than 200 hours.

Figure 2 shows the results of corrosion tests on 7075-T6 aluminum as a function of pH (controlled by NaOH or HCl additions) and indicates a minimum in corrosion rate in the pH 6-8 range. This data is similar to that reported for pure aluminum[10] in $H_2O + H_2SO_4$ or NaOH. Except that for the pure metal, the lowest corrosion rate has been reported to approach zero and, in general to be considerably lower for all pH's.

Figure 3 shows the S-N behavior for the alloy in a neutral

solution and at pH 2 and 10.2 where the overall corrosion rates

are approximately equal (about 100mdd). The data of Fig. 3 show

that there is not a direct correlation of fatigue behavior with

overall corrosion rate, pH 2 solutins reducing the 10^7 cycle

fatigue limit to a value of about 15MPa. Additionally, there

is a divergence of the data at higher applied stresses with the

pH 10.2 solution showing longer lines than either the pH 6

solution or the pH 2 solution. At low applied cyclic stresses

the pH 6 and pH 10.2 solutions produce approximately the same

fatigue resistance while the pH 2 solution shows an almost

constant percentage decrease in fatigue resistance when compared

to the neutral solution. In an earlier paper, it was suggested

that the reduction in fatigue resistance of high strength

aluminum alloys was associated with hydrogen (which was produced

by selective corrosion in the vicinity of non-metallic inclusions).

The pH results also suggest a hydrogen interaction since pH 2

solutions result in lower fatigue resistance. In order to test

this hypothesis, fatigue curves were generated for specimens

which were maintained at a potential of about 1300 versus the

saturated calomel electrode (about 600 mv cathodic to the

corrosion potential). The cathodic current density at this

potential corresponded to ≈ 0.7 ma/cm^2 which could be expected

to produce $\approx 10^{15}$ molecules H_2/cm^{2}sec. (assuming that the measured

current is only used to generate H_2). Figure 4 shows that, at
high applied cyclic stresses cathodic charging results in an
increase in fatigue resistance, while at lower applied stresses,
cathodic charging results in decreased fatigue resistance with
the 10^7 cycle fatigue limit reduced to a value of only $\approx$ 13 MPa.
Charging at lower potentials does not appreciably affect fatigue
resistance, although charging at higher potentials (less hydrogen
evolved) results in some cathodic protection, a maximum in
protection being observed at -900 mv vs. the saturated calomel
electrode. Anodic polarization results in a still larger reduction
in fatigue resistance (Fig. 5). The addition of a hydrogen atom
recombination poison (10 ppm As) to the sodium chloride solution
also results in a small reduction in fatigue resistance (Fig. 6).

Cathodic charging of the alloy exposed to neutral 0.5$\underline{N}$ Na_2SO_4
results in a large reduction in fatigue resistance in a similar
manner to cathodic charging in NaCl solution (Fig. 7). At
stresses in the vicinity of the 10^7-cycle fatigue limit, cathodic
charging in sulfate results in virtually the same fatigue resistance
as is observed in chlorides. At higher stresses, however sulfates
are slightly less aggressive than chlorides.

In addition to experiments on the 7075 alloy in the T6
condition, a limited number of experiments were conducted on
specimens in the T73 condition, a heat-treatment which has been
shown to be more resistant to stress corrosion cracking. Figure 8
shows the results of these experiments with an expected slight

slight decrease in fatigue resistance in dry air (YS and UTS
are lower for T73 than they are for the T6 heat treatment). In
aerated 0.5$\underline{N}$ NaCl solution, however, there is no appreciable
difference in fatigue resistance.

SEM fractography of specimens exposed to dry air, free
corrosion in NaCl and under cathodic charging conditions are
shown in Fig. 9. As has been previously reported[8], in dry air
the fracture surface consists of feathery river lines extending
from a single initiation site at the alloy surface consistent
with crystallographic (Stage I) cracking. In NaCl solution a
featureless, thumbnail-shaped area which is normal to the tensile
axis is associated with the initiation site. As the crack grows
out from this area, straight cleavage-like markings develop which
are unlike the feathery river lines observed in dry air in that
they are straighter and more well defined. These markings are
followed by the more traditional river line structure as the
crack progresses, although the striations which are observed at
longer crack lengths are of the "brittle" type observed by
Forsyth[2] in contrast to "ductile" striations observed in air.
In Na_2SO_4 solutions, the featureless region and the cleavage like
area is smaller than that observed for NaCl solutions (Fig.
10-a) "ductile" striations are observed near the overload
failure (Fig. 10-b). The featureless and cleavage like zones
observed after failure in solutions of pH 2 and pH 10.2 are

similar to those observed in neutral solutions (Fig. 11). Surface

pitting, associated with inclusions, was also observed in the

pH 2 solution although no secondary cracks were observed to be

associated with pits (Fig. 12). Cathodically polarizing specimens

to -1300mv vs SCE in NaCl solutions also resulted in the production

of a featureless plus cleavage zone and, additionally a significant

amount of Stage II, or non-crystallographic crack propagation was

observed in regions far from the crack initiation site (Fig. 13-a).

Polarization to -900mv vs. SCE also resulted in some grain

boundary attack in regions removed from the initiation site

(Fig. 13-b). Additionally, for cathodic potentials less than -900mv

vs. SCE in either NaCl or Na_2SO_4, extensive secondary cracking

and associated pitting was observed on alloy surfaces (Fig. 14-a).

Optical metallography of cross-sections of the free surfaces

indicated that the pits were associated with the cracks rather than

being responsible for their formation due to stress concentrations,

since sharp cracks also intersected the free surface adjacent to

cracks which were associated with pits. Presumably these cracks

formed late in specimen life since they are not extensively pitted

(Fig. 14-b and c). Additionally, the blunted nature of the

cracks on the specimen surface suggests that the cracks were

broadened by a corrosion reaction.

Discussion

The results of this study show that hydrogen, either produced by cathodic charging or by corrosion reactions, lowers the resistance of 7075 Al to cyclic deformation. Additionally, it has been shown that, under conditions of free corrosion, the anionic species is important to the fatigue process, Cl^- being considerably more damaging than SO_4 of the same ionic concentration. The application of cathodic currents in either Cl^- or $SO_4^=$ solution on the other hand results in a similar reduction in fatigue resistance, particularly at applied stresses in the vicinity of the 10^7-cycle fatigue limit. It has also been shown that a cathodic poison (As) reduces fatigue resistance in Cl^- solutions and that acid solutions are more damaging than basic solution of the same aggressiveness (as measured by overall corrosion rate).

Correlation of the fatigue resistance results with electron fractography and optical metallography indicates that hydrogen induces a form of cyclic cleavage in high strength aluminum alloys which is characterized by crack initiation on a plane normal to the tensile axis. This zone, which is essentially featureless, transforms to a zone of quasi-cleavage with a considerably more brittle appearance than is observed when fatigue crack propagation occurs in laboratory air. This region transforms to the feathery river line appearance similar to that of cracks propagated in air as the crack grows.

The featureless and quasi-cleavage zones are observed under all conditions of environmental fatigue, including tests performed at pH 2, pH 10.2 and under conditions of a small applied anodic potential. It should be pointed out, however, that even at pH 10.2, the corrosion potential of the alloy is more active than the reversible hydrogen electrode potential (φ_{corr} = -.96v_{SHE}, φ_{H_2} = -0.6v vs. SHE. Thus, thermodynamically, atomic hydrogen can be generated even at this high pH. Additionally, after the crack has initiated, it is likely that the pH within the growing crack is actually more acid than the bulk solution, further providing H_2 to the crack tip.

It is interesting to note that, in sulfate, externally generated hydrogen (in contrast to H_2 generated from an intrinsic corrosion reaction) results in almost the same fatigue resistance as is observed in Cl^- solutions. At relatively high stress levels, cathodic charging in the sulfate solution shows a slightly superior fatigue resistance while at stresses near the 10^7 cycle fatigue limit fatigue resistance is virtually identical. Since Cl^- is known to attack the protective film formed on Al alloys in neutral environments, it is likely that, at high stresses the film damage allows better access of the hydrogen to the alloy and accordingly lowers the crack initiation time. At low stresses, crack initiation time and initial crack propagation rates are slow so that there is ample time for the hydrogen to diffuse into the

surface of the alloy and to the tip of a small growing crack

without necessitating film damage by Cl^-. This hypothesis is

further supported by the observation that cathodic charging at

high stresses in the $SO_4^=$ solution does not affect fatigue

resistance (relative to free corrosion in $SO_4^=$), probably because

the crack propagation rate is sufficiently large to prevent an

equivalent H_2 concentration in the vicinity of a growing crack tip.

In chloride solutions, on the other hand, there is a slight

improvement of fatigue resistance at high stresses and a reduction

at lower stresses. Since it is believed that, while free corrosion

is occurring, hydrogen is evolved in the vicinity of preferentially

corroded pits and in a growing crack [11], the increased resistance

at high stresses may be due to a reduction in H_2 in a growing

crack due to some anodic polarization of the crevice created by

the growing crack. At low stresses, crack initiation and early

propagation are the limiting rate factors and the excess hydrogen

produced by cathodic polarization serves to shorten the time

required for these processes.

It is interesting, although probably not surprising, to note

that the T73 heat-treatment does not improve corrosion fatigue

resistance in Cl^- resistance. Since the T73 heat-treatment was

developed to provide intergranular SCC resistance by altering

grain boundary area structures, while the corrosion fatigue process

is transgranular, no beneficial effect should be expected.

The fractography results support the view that a form of hydrogen embrittlement is responsible for the reduction in fatigue resistance observed in this study since the featureless and cleavage like zones could result from local embrittlement of the alloy.

It has been suggested that aluminum alloys should not be expected to be susceptible to hydrogen embrittlement because of the low diffusivity of hydrogen in aluminum. However, the effect of hydrogen on fatigue behavior of aluminum alloys does not depend strongly on bulk diffusivity of hydrogen but only requires that hydrogen be present in the vicinity of the growing crack, i.e., in the plastic zone. In this region fresh metal is constantly being exposed to the environment. Additionally dislocations associated with the plastic zone may act as high diffusivity paths for hydrogen, thus locally increasing the hydrogen concentration[12,13]. At rapid crack propagation rates the crack would be expected to grow more rapidly than a "critical" (as yet undefined) concentration of hydrogen can be developed in the vicinity of the crack tip, and the fracture surface appearance will resemble that observed in air. An additional factor is that it has been shown that cathodic charging of a surface containing a crack or crevice can result in anodic polarization of the metal or alloy in the defect[11]. Thus less hydrogen would be available for embrittlement as the crack becomes longer. This suggestion may explain the constant life for decreasing potentials below some critical value, since the production of

hydrogen at the surface would not greatly affect conditions in the crack at long distances from the free surface.

The specific mechanisms of embrittlement is still not completely understood, although it may be associated with a reduction in plasticity at the crack tip by a Cottrell atmosphere phenomenon. For example, recent tensile tests conducted on a high purity analogue of the 7075 alloy showed serrated yielding of cathodically polarized specimens, suggesting a dislocation-solute interaction[12]. This observation, of a hydrogen dislocation interaction, also supports the hypothesis that dislocations in the plastic zone ahead of a growing crack may act as high diffusivity paths for hydrogen.

Summary and Conclusions

1. Neutral sulphate solutions under free corrosion conditions affect corrosion fatigue of 7075 Al alloys to approximately the same degree as distilled H_2O. Both solutions lower fatigue resistance when compared to laboratory air. Neutral chloride solutions are considerably more aggressive under free corrosion conditions.

2. Corrosion fatigue behavior of 7075 Al is not directly coupled to general corrosion rate, acid solutions reducing fatigue resistance more than basic solutions of the same corrosivity.

3.   Cathodic charging of 7075 Al results in a dramatic reduction in fatigue resistance in either sulfate or chloride solutions. This observation, coupled with fractographic evidence of cyclic cleavage, indicates that a form of hydrogen embrittlement is responsible for corrosion fatigue of this alloy.

4.   There is no inherent advantage to the T73 temper for resistance to corrosion fatigue in smooth surface specimens.

The results of this investigation suggest that dissolved hydrogen need only interact with the surface at nascent cracks and in the plastic zone of growing cracks, which accounts for the embrittlement effect in cyclic deformation experiments which is not observed in smooth surfaced, static deformation experiments.

Acknowledgments

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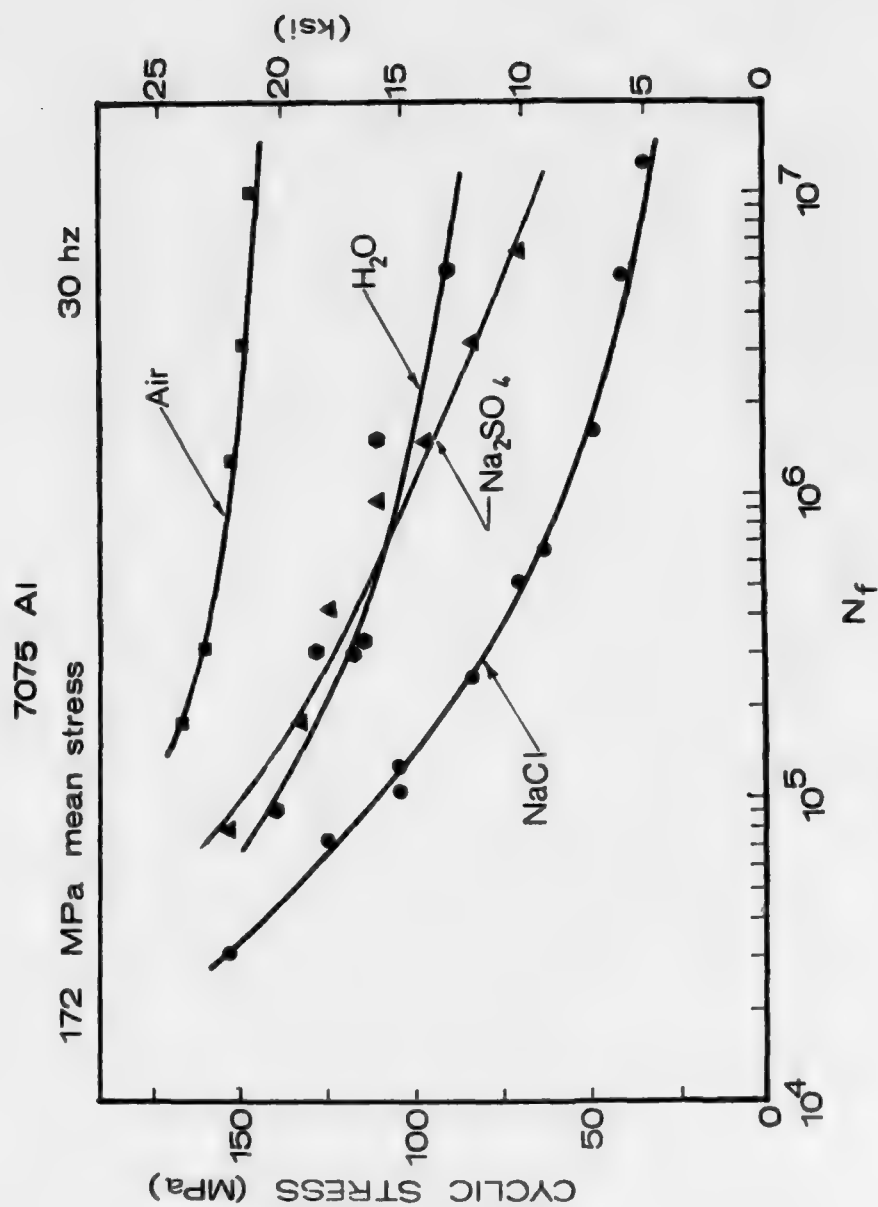
List of Figures

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Fig. 1 Fatigue behavior of 7075-T6 Al alloy in air, distilled H_2O and aqueous solutions of 0.5N Na_2SO_4 and 0.5N NaCl.
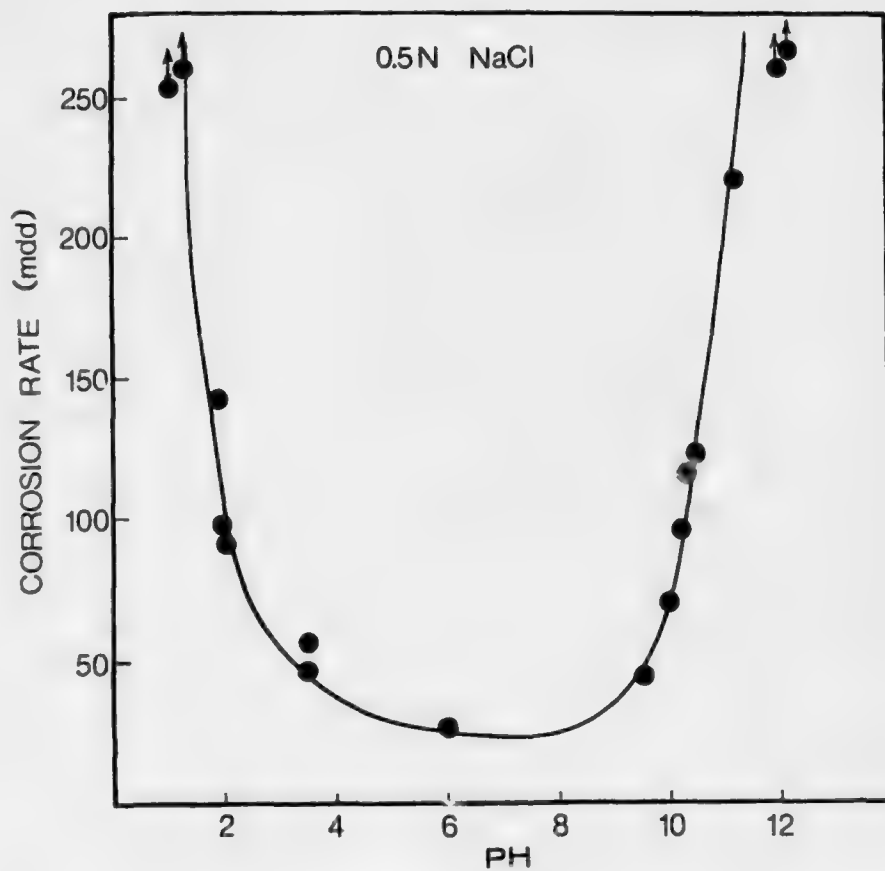
Fig. 2   The effect of pH on general corrosion of unstressed
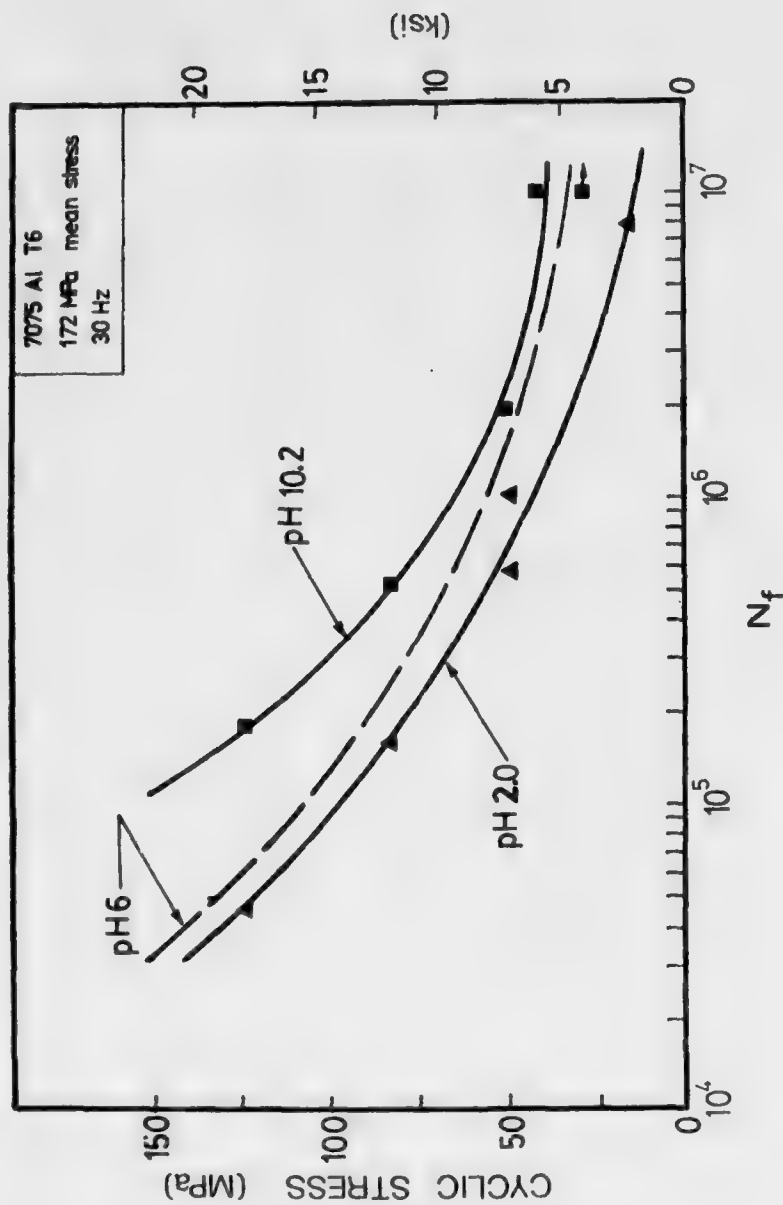7075-T6 alloy in aqueous 0.5N̲ NaCl solution (1mdd =
2cm/yr overall penetration).

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Fig. 3 Fatigue behavior of 7075-T6 Al in 0.5N NaCl solutions of pH2 and pHl0.2; pH6 data is repeated from Fig. 1.
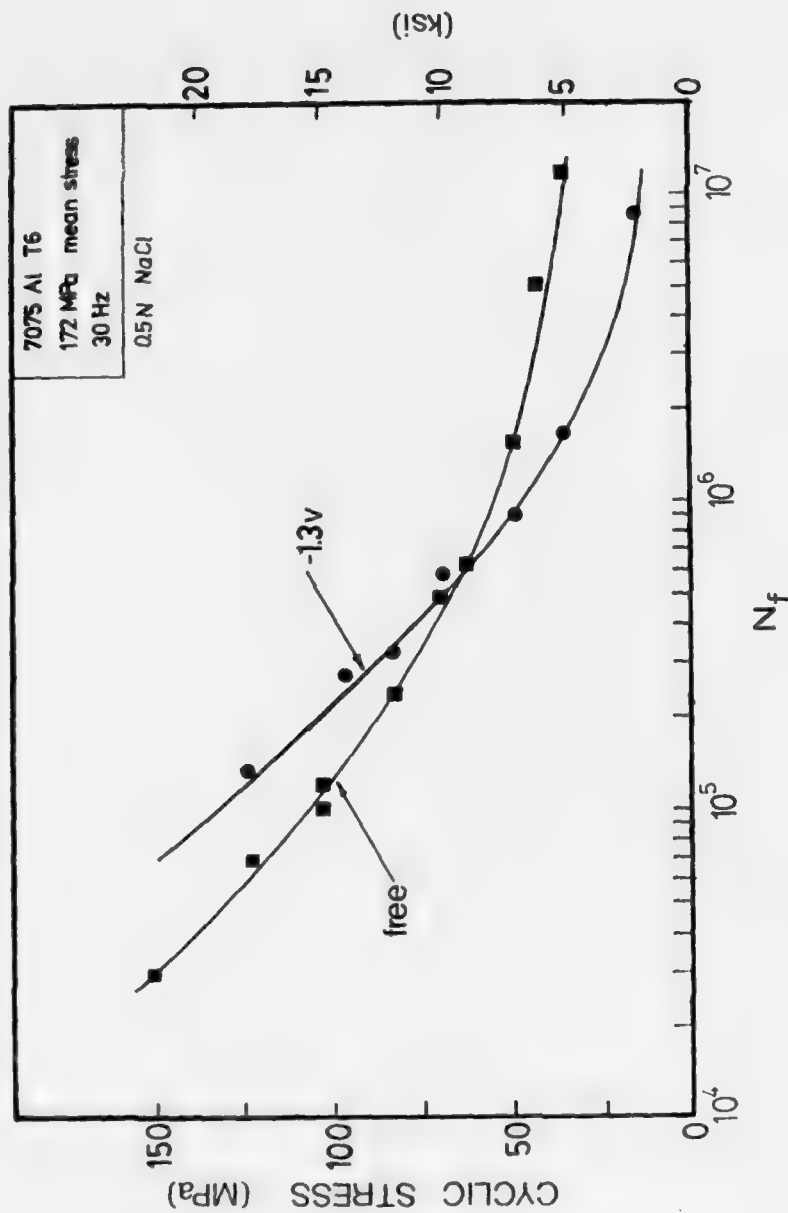
18

Fig. 4 Fatigue behavior of 7075-T6 Al in 0.5N NaCl under conditions of cathodic charging at φ_c= -1300 mv vs.SCE.

19
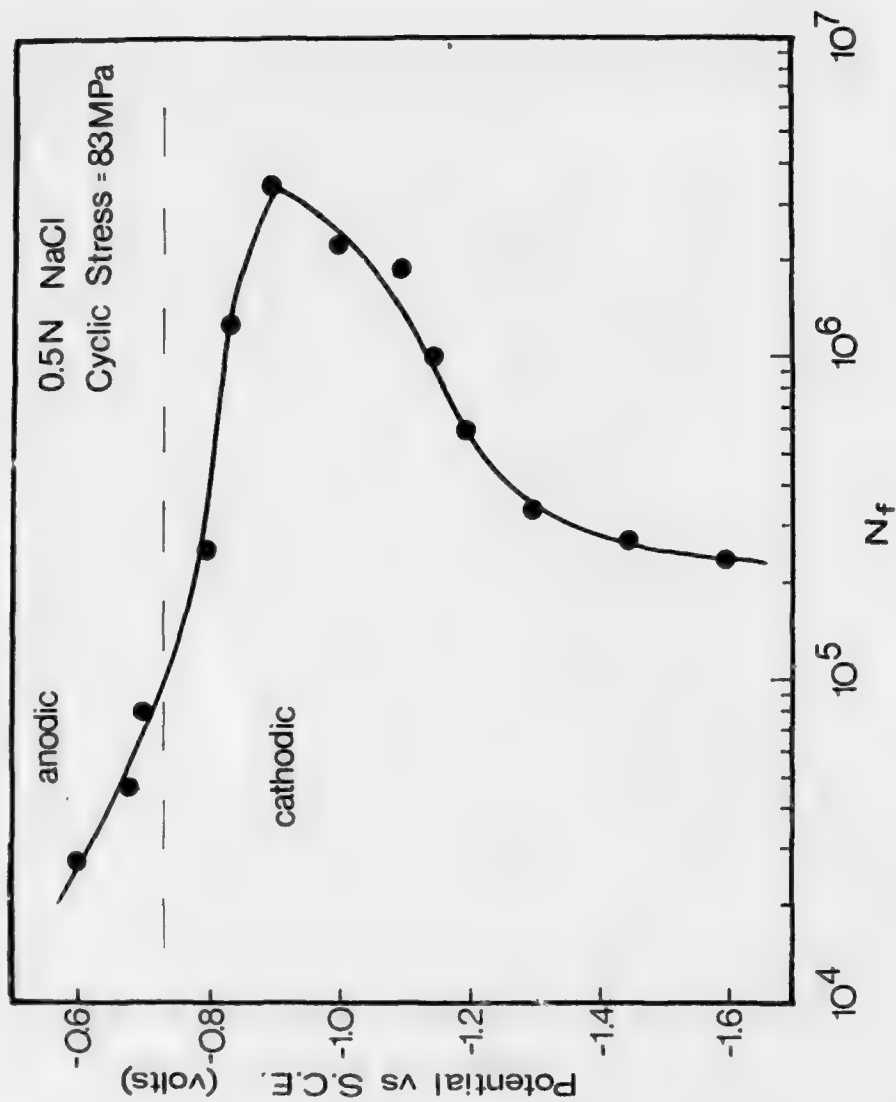
Fig. 5 Fatigue life of 7075-T6 Al in 0.5N NaCl under conditions of cathodic charging, σ_{max} = 255 MPa.

20
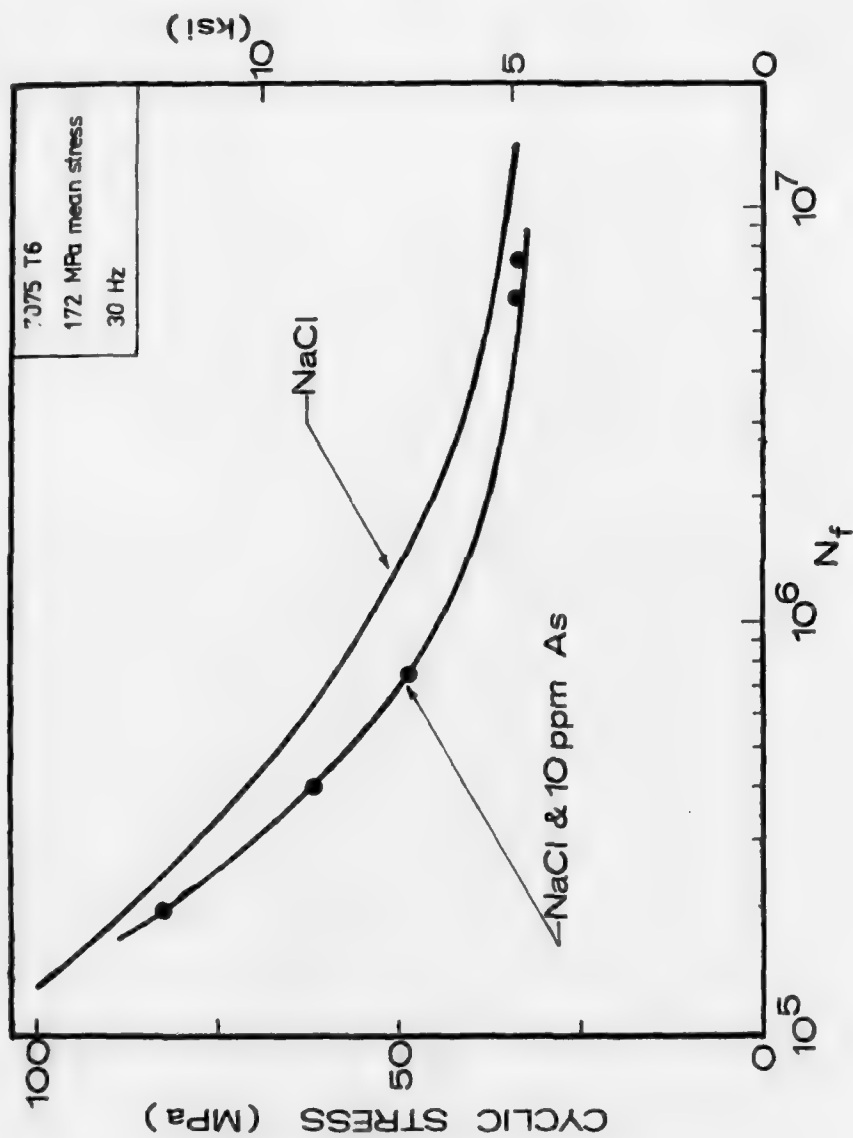
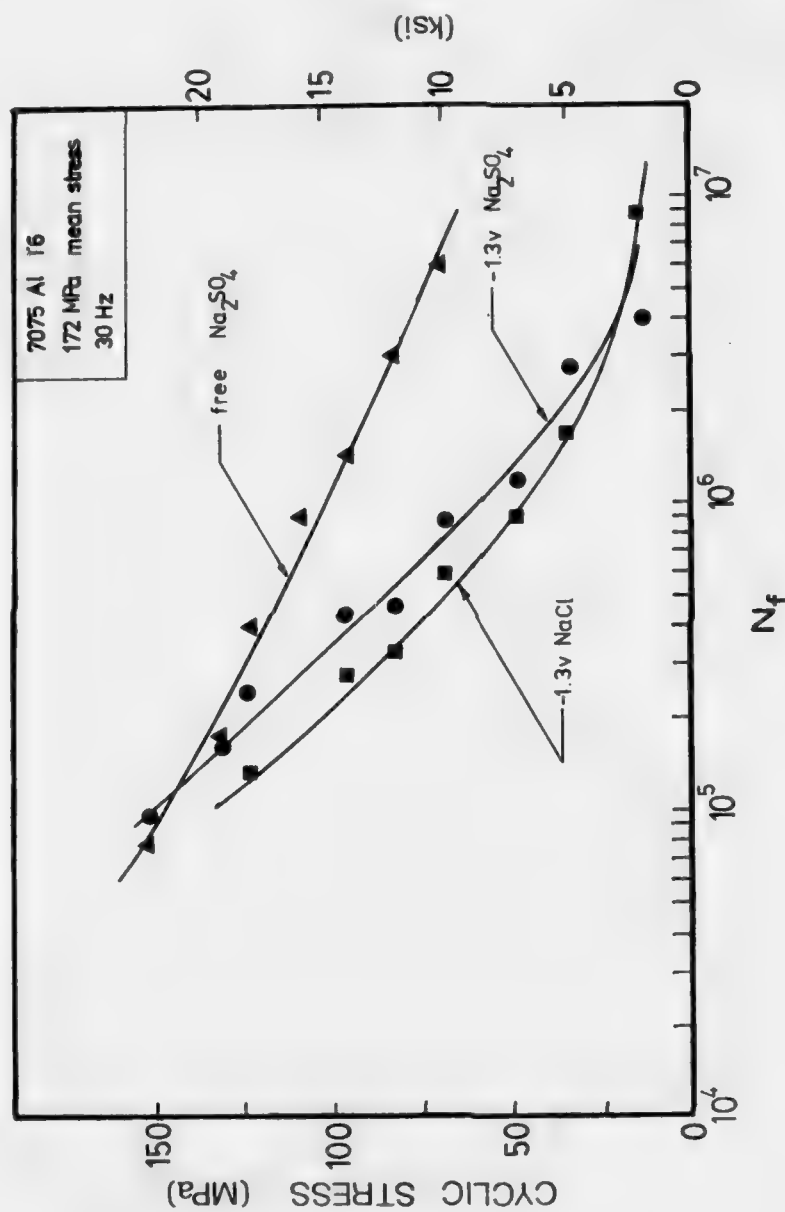Fig. 6 Fatigue behavior of 7075-T6 Al in 0.5N NaCl + 10ppm As.

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Fig. 7 Fatigue behavior of 7075T6 Al in 0.5N Na₂SO₄ under conditions of cathodic charging at φ_c= -1300 mv vs. SCE compared to free corrosion in 0.5N Na₂SO₄ and under conditions of cathodic charging in 0.5N NaCl at φ_c= -1300 mv vs. SCE.
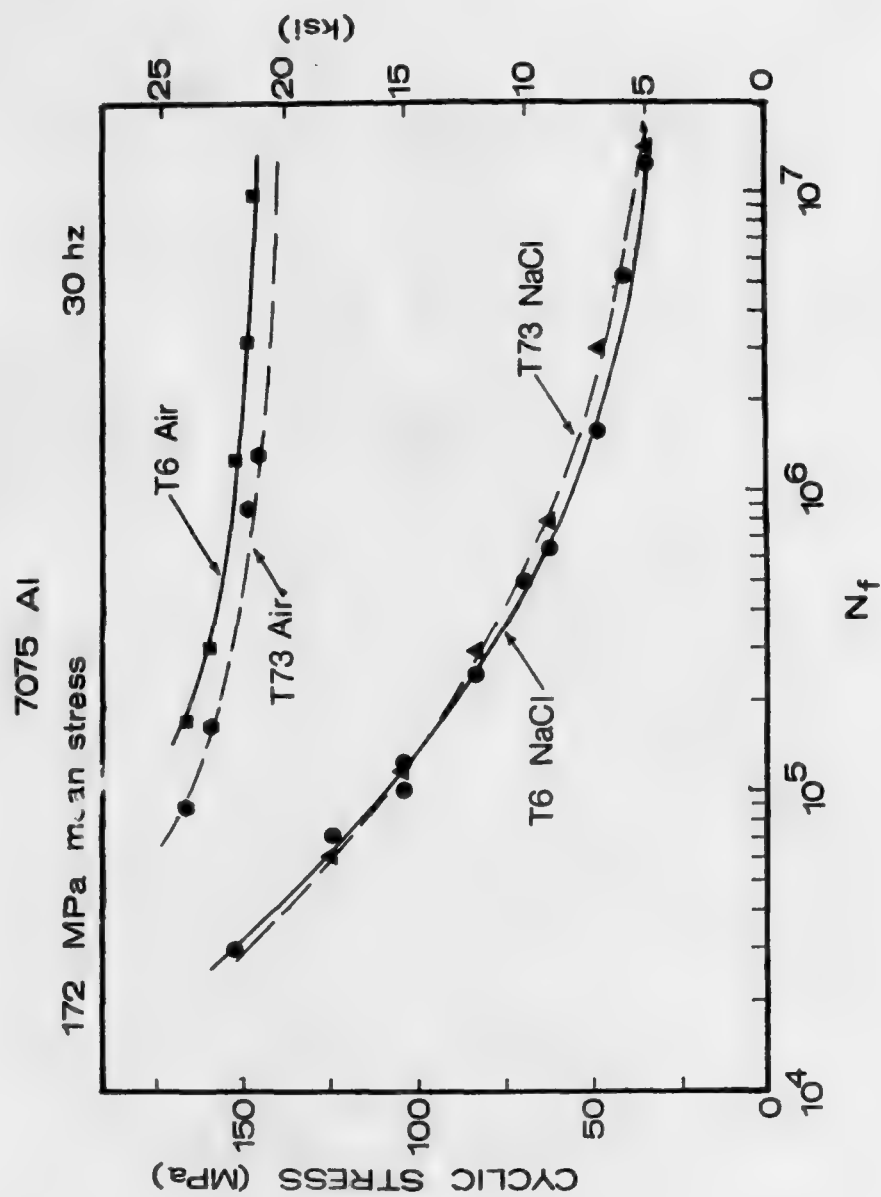
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Fig. 8 Fatigue behavior of 7075-T73 Al in air and in
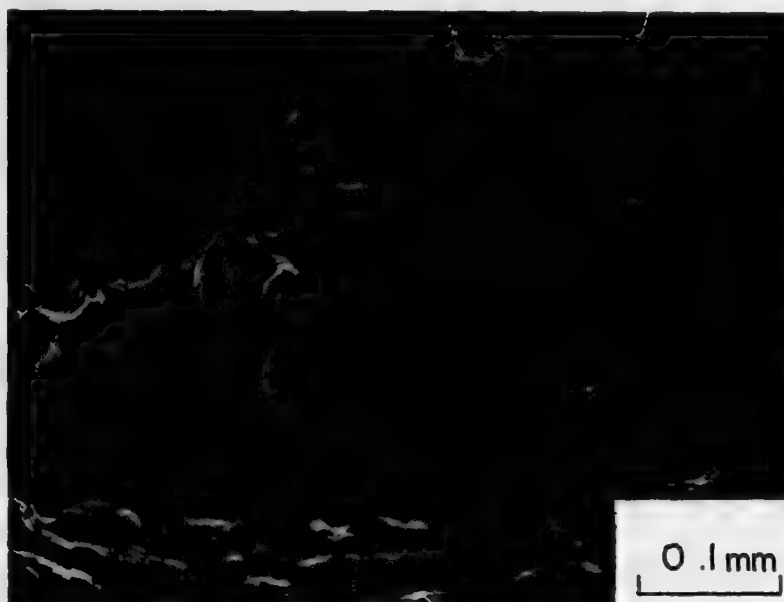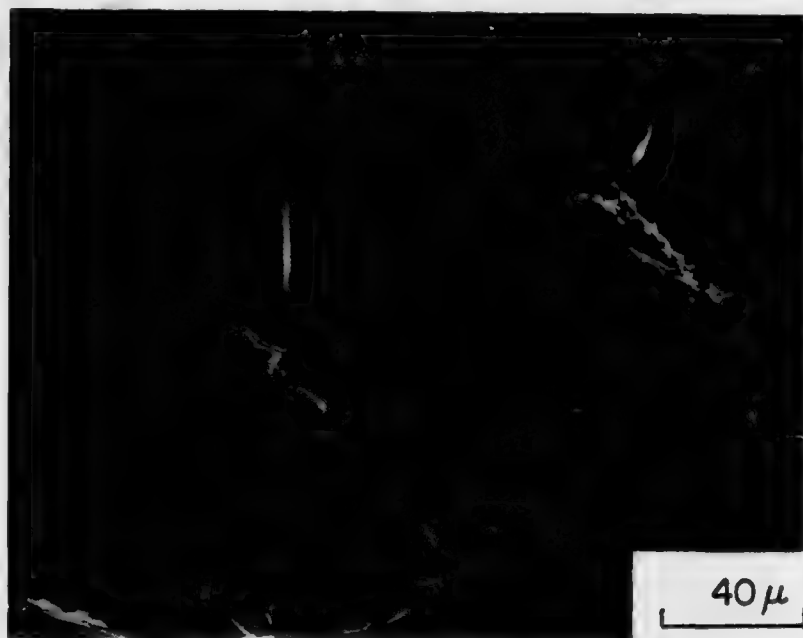0.5N NaCl compared to behavior of 7075-T6 Al.

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(a)

(b)

Fig. 9 Fracture surfaces of 7075-T6 Al in (a) air, (b)
freely corroding 0.5$\underline{N}$ NaCl solution and (c) 0.5$\underline{N}$
NaCl solution under conditions of cathodic
charging at φ_c= -1300 mv vs. SCE, $N_f = 10^6$.

24

0 .1 mm

(c)

25

(a)

40μ

(b)

10μ

Fig. 10 Fracture surface of 7075-T6 Al in 0.5$\underline{N}$ Na$_2$SO$_4$ solution $N_f = 5 \times 10^5$.

26

(a)

(b)

Fig. 11 Fracture surfaces of 7075-T6 Al in 0.5$\underline{N}$ NaCl of
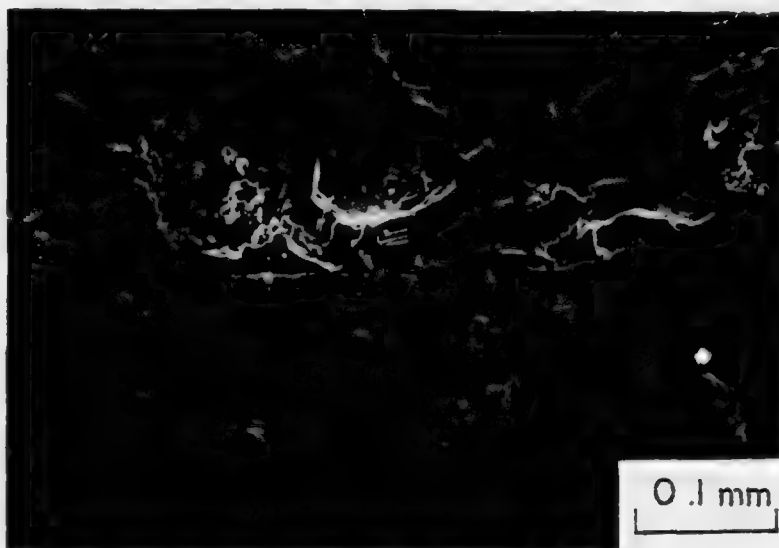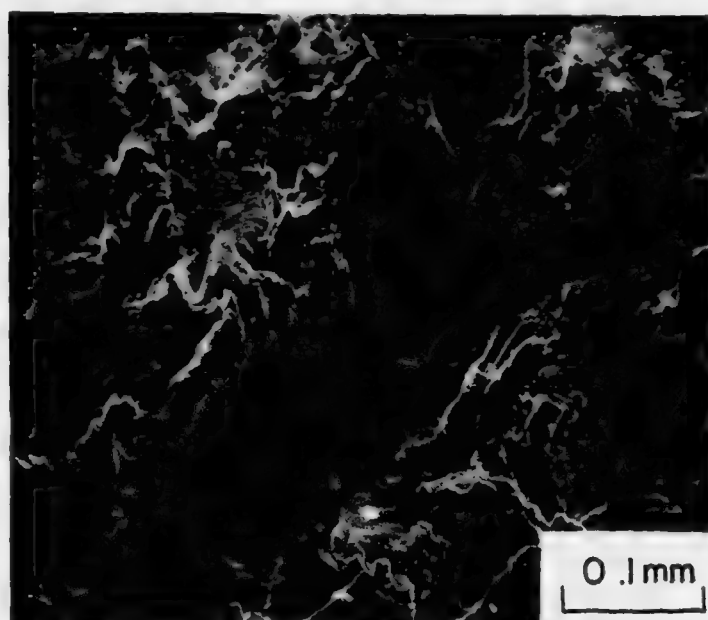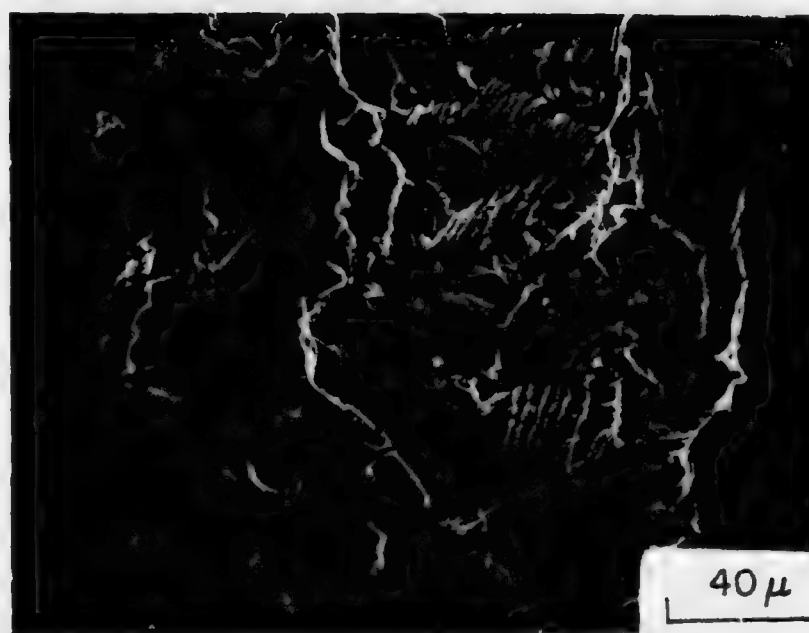(a) pH 2 and (b) pH10.2, $N_f = 10^6$.

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Fig. 12 Surface pitting of 7075-T6 Al in 0.5$\underline{N}$ NaCl solution of pH2.
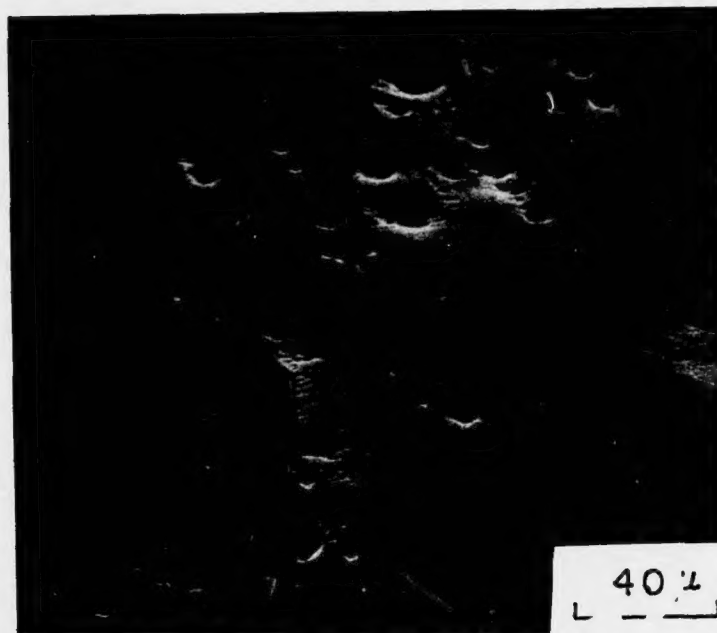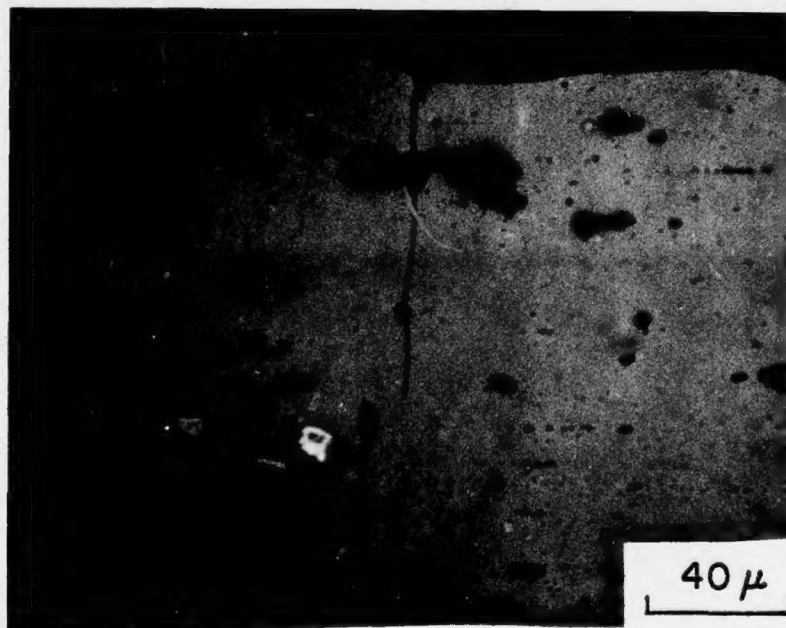
(a)

(b)

Fig. 13 Fracture surface of 7075-T7 Al in 0.5$\underline{N}$ NaCl under
conditions of cathodic charging (a) φ_C= -1300 mv
vs. SCE showing Stage II cracking and (b) φ_C=
-900 mv vs. SCE showing intergranular corrosion.

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(a)

(b)

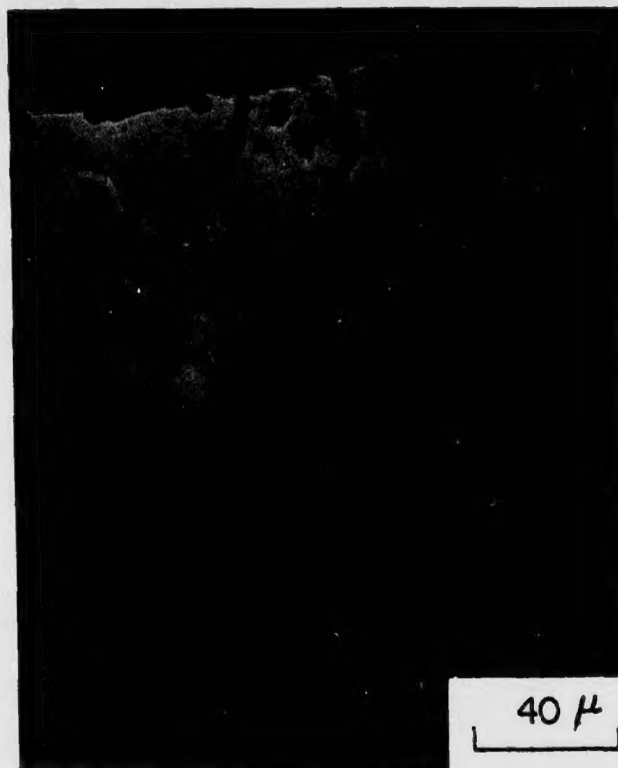Fig. 14   (a) Secondary cracking and pitting of 7075-T6 Al
         alloy in 0.5$\underline{N}$ NaCl under conditions of cathodic
         charging at φ_c= -1300 mv vs. SCE, (b and c) optical
         micrographs of a cross-section of 7075-T6 alloy
         showing secondary cracking and pits associated with
         secondary cracks under cathodic charging conditions.

30

(c)

40 μ

31

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13. ABSTRACT

High cycle fatigue experiments have been performed on a 7075 Al alloy, principally in the T6 temper in dry air, distilled water 0.5N NaCl and 0.5N Na_2SO_4 solutions as functions of pH, cathodic charging and catalyst poisoning of the hydrogen evolution reaction. All aqueous solutions appreciably lowered fatigue resistance with Cl^- ion producing the greatest reduction in resistance and $SO_4^=$ ion behaving essentially in the same manner as distilled water. Under cathodic charging conditions fatigue resistance is significantly reduced and both Cl^- and $SO_4^=$ solutions produce similar fatigue lives. Acid solutions reduce fatigue resistance to a greater degree than do basic solutions of equivalent corrosivity and a catalyst poison (As) added to Cl^- solutions reduces fatigue resistance relative to neutral Cl^- solutions. Fractography of specimens fatigued in aqueous environments shows that a significant amount of cleavage and quasi-cleavage occurs, the extent of these features being apparently a function of available hydrogen to the alloy free surface and to the tips of growing cracks. On the basis of these observations, it is suggested that corrosion fatigue of 7075 alloy is essentially a hydrogen embrittlement phenomenon where the low diffusivity of hydrogen is counterbalanced by the fact hydrogen need only be present in the alloy free surface for crack initiation and in the plastic zone of growing cracks for propagation.

14	KEY WORDS	LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
	embrittlement						
	fatigue resistance						
	hydrogen						
	cyclic stressing						